Supporting Information for

Bio-renewable Enantioselective Aldol Reaction in Natural Deep Eutectic Solvents.

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General Information: All reactions were carried out under argon. All reagents are commercially available and used without further purification. \(^1\)H NMR (300 MHz, 400 MHz) and \(^{13}\)C NMR (75 MHz) spectra were obtained at 25 °C using CDCl\(_3\) as solvent and chemical shifts are reported as δ values relative to TMS as internal standard. HPLC analyses were performed on equipped with a chiral column and automatic inyector, using mixtures of n-hexane/isopropyl alcohol (IPA) as mobile phase, at 25 °C. Analytical TLC was performed on silica gel plates and the spots were visualized under UV light (λ=254 nm). For flash chromatography we employed silica gel 60 (0.040-0.063 mm). For recycling experiments, an Edwards T-station equipped with a diaphragm pump 75 was used for water evaporation.

General procedure for the preparation of DES: The corresponding solid components of the desired DES in the correct proportion were placed in a 50 mL round-bottom flask. The resulting mixture was heated to 80 °C (from 1 to 3 h) under argon atmosphere with stirring until a clear colourless liquid was obtained.

General procedure for the aldol reaction in deep eutectic solvent: To around 1 mL of the corresponding solvent in a vessel under argon atmosphere, L-proline (0.035 g, 30 mol%) and the corresponding aldehyde (1 mmol) were added. Then the source of nucleophile was charged (5 mmol for the case of acetone and propanal, 1 mmol for cyclohexanone, 2 for the other ketones). The reaction mixture was stirred under argon atmosphere for 24 h to 5 days (see Table 1 and 2, Scheme 3 and text) at room temperature. Then, 2 mL of water were added and the mixture was extracted with ethyl acetate (3 × 1 mL). The resulting organic phase was dried over anhydrous magnesium sulphate, and the solvent was evaporated under reduced pressure. The resulting crude material was purified by percolation through a small pad of silica gel with 1:1 ethyl acetate/hexane mixtures. In the case of using propanal, after extraction, the resulting organic phase was dried over anhydrous magnesium sulphate, and the solvent was evaporated under reduced pressure. The resulting crude was treated with sodium borohydride (5 mmol, 190 mg) in methanol (3 mL). The reaction mixture was stirred during 2 h at 0 °C. After reaction, phosphate buffer (2 mL) was added, and the mixture was extracted with ethyl acetate (3 × 1 mL). The resulting organic phase was dried over anhydrous magnesium sulphate, and the solvent was evaporated under reduced pressure. The resulting crude material was purified by percolation through a small pad of silica gel with 1:1 ethyl acetate/hexane mixtures.

Recover and reuse of the catalyst and DES: To the corresponding solvent [aprox 3 mL: D-glucose (2.7 g) and D/L-malic acid (2.1 g)] were placed in a vessel under argon atmosphere. L-proline (0.175 g) and the corresponding aldehyde (5 mmol) were added. Then the source of nucleophile was charged (25 mmol). The reaction mixture was stirred under argon atmosphere for 24 h. Then, 10 mL of water was
added and the resulting organic upper layer was collected through a pipette for the gram scale procedure. The resulting organic phase was dried over anhydrous magnesium sulphate, and the solvent was evaporated under reduced pressure. The resulting crude material was purified recrystallization from ethyl acetate/hexane mixtures. The aqueous layer was evaporated under reduced pressure. Water traces were eliminated for the residue using a high-vacuum membrane pump system over 24 hours. Then, the flask containing the DES and L-proline was charged with a new batch of aldehyde and acetone.

Spectra data of aldol products

**4-Hydroxy-4-(4-nitrophenyl)butan-2-one:**[1]

\[ \text{O} \quad \text{OH} \quad \text{NO}_2 \]

\(3a\)

\(^1^H\) NMR (300 MHz, CDCl\(_3\), 25 °C, TMS): \(\delta = 2.22\) (s, 3H), 2.86 (d, \(J(H,H) = 2.9\) Hz, 2H), 3.59 (d, \(J(H,H) = 3.3\) Hz, 1H), 5.27 (dd, \(J(H,H) = 2.9, 3.3\) Hz, 1H), 7.55 (d, \(J(H,H) = 8.8\) Hz, 2H), 8.21 ppm (d, \(J(H,H) = 8.8\) Hz, 2H). \(^{13}C\) NMR (75 MHz, CDCl\(_3\), 25 °C, TMS): \(\delta = 30.5, 51.4, 68.7, 123.6, 126.3, 147.3, 149.8, 208.2\) ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AS column at 254 nm (n-hexane/i-PrOH: 85/15, 1.0 mL/min), \(t_R = 16.8\) (major), \(t_R = 26.8\) (minor).

\[ \text{O} \quad \text{OH} \quad \text{NO}_2 \]

\(3b\)

**4-Hydroxy-4-(2-nitrophenyl)butan-2-one:**[2]

\(^1^H\) NMR (400 MHz, CDCl\(_3\), 25 °C, TMS): \(\delta = 2.17\) (s, 3H), 2.92-2.73 (m, 2H), 3.60 (br s, 1H), 3.82 (s, 3H), 5.41 (d, 1H, \(J(H,H) = 8\) Hz), 6.86 (d, 1H, \(J(H,H) = 8\) Hz), 6.97 (t, 1H, \(J(H,H) = 8\) Hz), 7.25 (t, 1H, \(J(H,H) = 8\) Hz), 7.44 ppm (d, 1H, \(J(H,H) = 8\) Hz). \(^{13}C\) NMR (100 MHz, CDCl\(_3\), 25 °C, TMS): \(\delta = 30.5, 50.3, 55.2, 65.3, 110.2, 120.7, 126.2, 128.3, 130.9, 155.7\) ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel ADH column at 254 nm (n-hexane/i-PrOH: 98/2, 1.0 mL/min), \(t_R = 41.7\) (major), \(t_R = 45\) (minor).

\[ \text{O} \quad \text{OH} \quad \text{NO}_2 \]

\(3c\)

**4-Hydroxy-4-(3-nitrophenyl)butan-2-one:**[2]
\[ ^1 \text{H NMR (300 MHz, CDCl}_3, 25 \degree \text{C, TMS)}: \delta = 2.21 (s, 3H), 2.69-2.96 (m, 2H), 3.39 (s, 1H), 5.12 (dd, 1H, J(H,H)=7.8, 4.5 Hz), 7.24 (d, 2H, J(H,H)= 8.3 Hz), 7.48 ppm (d, 2H, J(H,H)=8.4 Hz.). \]

\[ ^{13} \text{C NMR (75 MHz, CDCl}_3, 25 \degree \text{C, TMS)}: 30.7, 51.5, 68.8, 120.7, 122.6, 129.5, 131.8, 144.7, 148.3, 208.8 ppm. \]

The enantiomeric excess was determined by HPLC with a Chiralcel ADH column at 254 nm (\textit{n}-hexane/i-PrOH: 95/5, 1.0 mL/min), \( t_R = 21.4 \) (major), \( t_R = 22.7 \) (minor).

![4-(4-Cyanophenyl)-4-hydroxybutan-2-one](image)

\[ \text{4-(4-Cyanophenyl)-4-hydroxybutan-2-one:[2]} \]

\[ ^1 \text{H NMR (300 MHz, CDCl}_3, 25 \degree \text{C, TMS)}: \delta = 2.21 (s, 3H), 2.83 (m, 2H), 5.05-5.24 (m, 1H), 7.47 (d, 2H, J(H,H)= 8.7 Hz), 7.63 ppm (d, 2H, J(H,H)=8.7 Hz). \]

\[ ^{13} \text{C NMR (75 MHz, CDCl}_3, 25 \degree \text{C, TMS)}: 29.4, 51.6, 68.9, 111.2, 118.8, 126.4, 132.4, 148.1, 208.7 ppm. \]

The enantiomeric excess was determined by HPLC with a Chiralcel ODH column at 230 nm (\textit{n}-hexane/i-PrOH: 95/5, 1.0 mL/min), \( t_R = 31.2 \) (major), \( t_R = 36.3 \) (minor).

![4-Hydroxy-4-(4-(trifluoromethyl)phenyl)butan-2-one](image)

\[ \text{4-Hydroxy-4-(4-(trifluoromethyl)phenyl)butan-2-one:[2]} \]

\[ ^1 \text{H NMR (300 MHz, CDCl}_3, 25 \degree \text{C, TMS)}: \delta = 2.22 (s, 3H), 2.86 (d, J(H,H)=2.9 Hz, 2H), 3.59 (d, J(H,H)= 3.3 Hz, 1H), 5.27 (dd, J(H,H)= 2.9, 3.3 Hz, 1H), 7.55 (d, J(H,H)= 8.8 Hz, 2H), 8.21 ppm (d, J(H,H)= 8.8 Hz, 2H). \]

\[ ^{13} \text{C NMR (75 MHz, CDCl}_3, 25 \degree \text{C, TMS)}: \delta = 30.5, 51.4, 68.7, 123.6, 126.3, 147.3, 149.8, 208.2 ppm. \]

The enantiomeric excess was determined by HPLC with a Chiralcel AS column at 230 nm (\textit{n}-hexane/i-PrOH: 92/8, 1.0 mL/min), \( t_R = 8.6 \) (major), \( t_R = 10.8 \) (minor).

![4-(2-Chlorophenyl)-4-hydroxybutan-2-one](image)

\[ \text{4-(2-Chlorophenyl)-4-hydroxybutan-2-one:[1]} \]
$^1$H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): $\delta=2.17$ (s, 3H), 2.61-2.96 (m, 2H), 3.80 (br s, 1H), 5.46-5.55 (m, 1H), 7.14-7.28 ppm (m, 4H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): 30.5, 50.1, 66.5, 127.1, 127.2, 128.5, 129.5, 129.3, 209.0 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AS column at 254 nm ($n$-hexane/i-PrOH: 98/2, 1.0 mL/min), $t_R = 20.8$ (minor), $t_R = 24.4$ (major).

4-Hydroxy-4-phenylbutan-2-one:[1]

$^1$H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): $\delta=2.21$ (s, 3H), 2.83 (dd, 1H, $J$(H,H)=3.3, 17.7 Hz), 2.90 (dd, 1H, $J$(H,H)=9.0, 17.7 Hz), 5.16 (dd, 1H, $J$(H,H)=3.3, 9.0 Hz)), 7.29 ppm (m, 5H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): 30.8, 50.2, 69.8, 125.6, 128.6, 127.7, 128.5, 142.6, 209.3 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AS column at 210 nm ($n$-hexane/i-PrOH: 90/10, 1.0 mL/min), $t_R = 11.5$ (major), $t_R = 14.1$ (minor).

4-Hydroxy-4-(4-tolil)butan-2-one:[3]

$^1$H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): $\delta=2.20$ (s, 3H), 2.35 (s, 3H), 2.82 (d, $J$(H,H)=3.0 Hz, 2H), 3.33 (br s, 1H), 5.12 (d, $J$(H,H)= 3.0 Hz, 1H), 7.17 (d, $J$(H,H)= 8.9 Hz, 2H), 7.26 ppm (d, $J$(H,H)= 8.9 Hz, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): $\delta=21.1$, 30.8, 52.0, 69.7, 125.6, 128.2, 129.7, 137.4, 209.2 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel IA column at 280 nm ($n$-hexane/i-PrOH: 95/5, 1.0 mL/min), $t_R = 10.5$ (minor), $t_R = 12.6$ (major).

4-Hydroxy-4-(cyclohexyl)-butan-2-one:[3]
$^1$H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=0.95-1.25 (m, 6H), 1.61-1.76 (m, 5H), 2.18 (s, 3H), 2.53 (m, 2H), 2.89 (br s, 1H), 3.82 (m, 1H) ppm. $^{13}$C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=25.8, 26.2, 26.5, 28.1, 29.0, 30.7, 42.9, 47.7, 71.9, 210.7 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AS column at 210 nm ($n$-hexane/i-PrOH: 90/10, 1.0 mL/min), $t_R$ = 9.5 (major), $t_R$ = 11.3 (minor).

![Image of compound 3j]

2-[Hydroxy(4-nitrophenyl)methyl]cyclohexanone:[3]

$^1$H NMR (300 MHz, CDCl$_3$ CDCl$_3$, 25 °C, TMS): $\delta$=1.28-1.49 (m, 1H), 1.52-1.73 (m, 3H), 1.79-1.83 (m, 1H), 2.06-2.14 (m, 1H), 2.21-2.31 (m, 1H), 2.33-2.50 (m, 1H), 2.54-2.63 (m, 1H), 3.12 (br s, 1H syn), 4.02 (br s, 1H anti), 4.88 (d, $J$(H,H)=8.4 Hz, 1H anti), 5.46 (s, 1H syn), 7.49 (d, $J$(H,H)=8.7 Hz, 2H), 8.19 ppm (d, $J$(H,H)=8.7 Hz, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$25 °C, TMS): $\delta$=anti 24.6, 27.5, 30.6, 42.6, 57.1, 73.9, 123.5, 127.8, 147.4, 148.3, 214.6.

The enantiomeric excess was determined by HPLC with a Chiralcel ADH column at 254 nm ($n$-hexane/i-PrOH: 90/10, 1.0 mL/min), anti: $t_R$ = 19.1 (minor), $t_R$ = 25.1 (major), syn: $t_R$ = 15.0 (minor), $t_R$ = 17.1 (major).

![Image of compound 3k]

2-[Hydroxy(4-(trifluoromethyl)phenyl)methyl]cyclohexanone:[4]

$^1$H NMR (300 MHz, CDCl$_3$ CDCl$_3$, 25 °C, TMS): $\delta$=1.49-1.83 (m, 5H), 2.05-2.10 (m, 1H), 2.30-2.48 (m, 2H), 2.65-2.71 (m, 1H), 4.05 (d, $J$(H,H)=4.0 Hz, 1H), 5.35 (dd, $J$(H,H)=4.0, 8.2 Hz, 1H), 7.49 (d, $J$(H,H)=8.7 Hz, 2H), 8.19 ppm (d, $J$(H,H)=8.7 Hz, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=anti 24.8, 27.8, 30.3, 42.6, 57.5, 70.3, 127.1, 128.1, 128.6, 129.1, 132.8, 139.0, 215.1 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AD column at 210 nm ($n$-hexane/i-PrOH: 90/10, 1.0 mL/min), anti: $t_R$ = 20.5 (minor), $t_R$ = 26.3 (major), syn: $t_R$ = 13.9 (minor), $t_R$ = 16.2 (minor).
2-[(2-Chlorophenyl)hydroxymethyl]cyclohexanone:[4]  
$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC, TMS): $\delta=1.49$-$1.83$ (m, 5H), 2.05-$2.10$ (m, 1H), 2.30-$2.48$ (m, 2), 2.65-$2.71$ (m, 1H), 4.05 (d, $J$(H,H)=3.9 Hz, 1H), 5.35 (dd, $J$(H,H)=3.9, 8.1 Hz, 1H), 7.18-$7.22$ (m, 1H), 7.27-$7.34$ (m, 2H), 7.54-$7.56$ ppm (m, 1H).  
$^{13}$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): $\delta=anti$ 25.0, 27.8, 30.6, 42.7, 57.6, 72.9, 123.4, 127.9, 128.5, 129.1, 132.5, 140.7, 215.2 ppm.  
The enantiomeric excess was determined by HPLC with a Chiralcel ODH column at 280 nm ($n$-hexane/$i$-PrOH: 95/5, 1.0 mL/min), $anti: t_R = 8.5$ (major), $t_R = 10.8$ (minor), $syn: t_R = 6.4$ (minor), $t_R = 7.9$ (major).

2-[Hydroxy-(4-(fluorophenyl)methyl]-cyclohexanone:[5]  
$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC, TMS): $\delta=1.22$-$2.08$ (m, 6H), 2.31-$2.65$ (m, 3H), 4.03 (br s, 1H), 4.77 (d, $J$(H,H)=8.4 Hz, 1H), 7.03 (d, $J$(H,H)=8.7 Hz, 2H), 7.33 ppm (d, $J$(H,H)=8.7 Hz, 2H).  
$^{13}$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): $\delta=anti$ 24.6, 27.7, 30.7, 42.6, 57.4, 74.1, 115.2, 128.5, 136.5, 162.6, 215.4 ppm.  
The enantiomeric excess was determined by HPLC with a Chiralcel ADH column at 210 nm ($n$-hexane/$i$-PrOH: 90/10, 0.3 mL/min), $anti: t_R = 42.6$ (minor), $t_R = 47.3$ (major), $syn: t_R = 28.8$ (minor), $t_R = 32.9$ (major).

3-hydroxy(4-nitrophenyl)methyl)tetrahydro-4H-thiopyran-4-one:[6]  
$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC, TMS): $\delta=2.48$-$2.55$ (m, 1H), 2.65 (t, $J$(H,H)=12.2 Hz, 1H), 2.71-$2.84$ (m, 2H), 2.96-$3.05$ (m, 3H), 3.67 (br s, 1H), 5.05 (d, $J$(H,H)=7.9 Hz, 1H, $anti$), 5.52 (br s, 1H, $syn$),
7.55 (d, J(H,H)=8.8 Hz, 2H), 8.24 ppm (d, J(H,H)=8.8 Hz, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): δ=anti 30.7, 32.8, 44.7, 59.4, 73.1, 123.8, 127.7, 147.6, 147.7, 211.2 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AD column at 280 nm (n-hexane/i-PrOH: 90/10, 1.0 mL/min), anti: $t_R = 38.6$ (minor), $t_R = 69.2$ (major). syn: $t_R = 29.6$ (mayor), $t_R = 59.4$ (minor).

\[
\text{syn-3o}
\]

2-[Hydroxy(4-nitrophenyl)methyl]cyclopentanone:[3]

$^1$H NMR (300 MHz, CDCl$_3$ CDCl$_3$, 25 ºC, TMS): δ=□ 1.72-1.75 (m, 2H), 1.96-2.09 (m, 1H), 2.30-2.74 (m, 2H), 2.74 (d, J(H,H)=4.8 Hz, 1H, syn), 4.77 (br s, 1H, anti), 4.84 (d, J(H,H)=9.1 Hz, 1H, syn), 5.42 (s, 1H), 7.52 (d, J(H,H)=8.4 Hz, 2H), 8.21 ppm (d, J(H,H)=8.7 Hz, 2H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): δ=anti 20.2, 22.2, 38.8, 56.0, 70.3, 123.6, 126.3, 147.0, 150.2, 219.6; anti 20.2, 26.7, 38.5, 55.0, 74.3, 123.5, 127.3, 147.2, 148.5, 219.7 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel AD column at 280 nm (n-hexane/i-PrOH: 96/4, 1.0 mL/min), syn: $t_R = 31.4$ (major), $t_R = 46.0$ (minor). anti: $t_R = 55.9$ (minor), $t_R = 58.9$ (major).

\[
\text{anti-3p}
\]

2-(-1-hydroxyundecyl)cyclopentan-1-one:[7]

$^1$H NMR (300 MHz, CDCl$_3$ CDCl$_3$, 25 ºC, TMS): δ=□ 0.83 (t, J(H,H)=6.6 Hz, 3H), 1.23 (br s, 2H), 1.36-2.29 (m, 22H), 4.03 ppm (dt, J(H,H)=3, 6.6 Hz, 1H). $^{13}$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): δ=14.1, 20.6, 22.8, 26.0, 29.3, 29.4, 29.45, 29.5, 29.6, 31.8, 34.8, 39.1, 54.4, 96.5, 221.7 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel IA column at 254 nm (n-hexane/i-PrOH: 99/1, 1.0 mL/min), syn: $t_R = 8.6$ (minor), $t_R = 10.5$ (major). anti: $t_R = 14.8$ (major), $t_R = 20.6$ (minor).

\[
\text{anti-3q}
\]
3,4-Dihydroxy-4-(4-nitrophenyl)butan-2-one:[8]

$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC, TMS): $\delta$= 2.02 (s, 3H), 3.03 (d, $J$(H,H)=4.3 Hz, 1H), 3.71 (d, $J$(H,H)=4.9 Hz, 1H), 4.60-4.91 (m, 1H), 5.08-5.11 (m, 1H), 7.62 (d, $J$(H,H)=8.4 Hz, 2H), 8.24 ppm (d, $J$(H,H)=8.9 Hz, 2H). $^1$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): $\delta$=27.7, 74.3, 80.5, 123.7, 127.0, 146.3, 147.3, 207.2 ppm.

The enantiomeric excess was determined by HPLC with a Chiralcel ADH column at 254 nm ($n$-hexane/i-PrOH: 80/20, 0.8 mL/min), $anti$: $t_R$ = 10.5 (minor), $t_R$ = 11.7 (major), $syn$: $t_R$ = 13.4 (mayor), $t_R$ = 17.1 (minor).

4-Hydroxy-3-methoxy-4-(4-nitrophenyl)butan-2-one:[8]

$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC, TMS): $\delta$= 2.16 (s, 3H), 3.20 (s, 1H), 3.32 (s, 3H), 3.70 (d, $J$(H,H)=6.2 Hz, 1H), 5.02 (d, $J$(H,H)=6.2 Hz, 1H), 7.56 (d, $J$(H,H)=8.8 Hz, 2H), 8.22 ppm (d, $J$(H,H)=8.8 Hz, 2H). $^1$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): $\delta$=27.5, 59.6, 73.3, 89.6, 123.4, 127.7, 146.7, 147.7, 209.9 ppm.

The enantiomeric excess was determined by HPLC with a Chiralpak ODH column at 280 nm ($n$-hexane/i-PrOH: 90/10, 0.8 mL/min), $t_R$ = 12.9 (major), $t_R$ = 15.7 (minor).

2-Methyl-1-(4-nitrophenyl)propane-1,3-diol:[9]

$^1$H NMR (300 MHz, CDCl$_3$, 25 ºC, TMS): $\delta$= 0.78 (d, $J$(H,H)=7.0 Hz, 3H), 2.01-2.06 (m, 1H), 2.74 (br s, 1H), 3.72-3.85 (m, 3H), 4.72 (d, $J$(H,H)=7.8 Hz, 1H $anti$), 7.54 (d, $J$(H,H)=8.7 Hz, 2H), 8.23 ppm (d, $J$(H,H)=8.7 Hz, 2H). $^1$C NMR (75 MHz, CDCl$_3$, 25 ºC, TMS): $\delta$=13.6, 41.5, 67.4, 79.3, 123.6, 127.5, 147.4, 150.5 ppm.

The enantiomeric excess was determined by HPLC with a Chiralpak AD column at 210 nm ($n$-hexane/i-PrOH: 97/3, 1.0 mL/min), $anti$: $t_R$ = 89.6 (major), $t_R$ = 94.5 (minor); $syn$: $t_R$ = 79.3 (major), $t_R$ = 85.3 (minor).
2-Methyl-1-(2’-chlorophenyl)propane-1,3-diol:[10]

$^1$H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=0.87 (t, $J$(H,H)=7.2 Hz, 3H), 2.10-2.15 (m, 1H), 2.59-2.62 (m, 1H), 3.09 (d, $J$(H,H)=3.9 Hz, 1H), 3.72-3.78 (m, 2H), 5.13 (dd, $J$(H,H)=3.8 Hz, 7.2 Hz, 1H), 7.17-7.24 (m, 1H), 7.30-7.35 (m, 2H), 7.58-7.61 ppm (m, 1H). 13C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=13.6, 40.6, 67.4, 76.1, 127.1, 128.0, 128.6, 129.4, 132.4, 140.8 ppm.

The enantiomeric excess was determined by HPLC with a Chiralpak AD column at 230 nm ($n$-hexane/i-PrOH: 97/3, 1.0 mL/min), $\text{anti}$: $t_R$ = 39.1 (major), $t_R$ = 60.5 (minor); $\text{syn}$: $t_R$ = 23.8 (major), $t_R$ = 32.7 (minor).

2-Methyl-1-(4-trifluromethylphenyl)propane-1,3-diol:[10]

$^1$H NMR (300 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=0.72 (d, $J$(H,H)=7.0 Hz, 3H), 2.01-2.03 (m, 1H), 2.89 (br s, 1H), 3.66-3.79 (m, 3H), 4.61 (d, $J$(H,H)=7.9 Hz, 1H $\text{anti}$), 5.04 (br s, 1H, $\text{syn}$) 7.45 (d, $J$(H,H)=8.1 Hz, 2H), 7.61 ppm (d, $J$(H,H)=8.1 Hz, 2H). 13C NMR (75 MHz, CDCl$_3$, 25 °C, TMS): $\delta$=13.6, 41.4, 67.6, 79.9, 125.2, 126.9, 130.0, 147.2.

The enantiomeric excess was determined by HPLC with a Chiralpak AD column at 230 nm ($n$-hexane/i-PrOH: 97/3, 1.0 mL/min), $\text{anti}$: $t_R$ = 28.5 (major), $t_R$ = 30.3 (minor); $\text{syn}$: $t_R$ = 18.3 (major), $t_R$ = 19.9 (minor).
NMR spectra for aldol products

3a

3b

S11
anti-3j
anti-31
anti-5b
HPLC for aldol products

\[
\text{RAC-3a}
\]

AS, 85:15, 1mL

Injection Date : 29/04/2015 20:34:44
Sample Name : RG1612
Acq. Operator : RG
Acq. Instrument : HPLC-GPC
Acq. Method : C:\HPCEM\1\METHODS\RG.M
Last changed : 29/04/2015 20:33:47 by RG
Analysis Method : C:\HPCEM\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Signal 1: DAD1 A, Sig=254.4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width [min]</th>
<th>Area [mAU*sec]</th>
<th>Height [mAU]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.6946</td>
<td>5.35379e4</td>
<td>1040.03723</td>
<td>48.4599</td>
</tr>
<tr>
<td>2</td>
<td>0.9978</td>
<td>5.69409e4</td>
<td>675.26349</td>
<td>51.5401</td>
</tr>
</tbody>
</table>
AS, 85:15 (Hx:iPrOH), 1.0 mL/min, HPLC2

Injection Date : 30/03/2016 10:35:13  Seq. Line : 1
Sample Name : RG1578C1  Location : Vial 11
Acq. Operator : RG  Inj : 1
Acq. Instrument : HPLC 2  Inj Volume : 7 μl
Acq. Method : C:\HPChem\2\METHODS\C3_1.60.M
Last changed : 30/03/2016 10:34:56 by RG
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\C4.M
Last changed : 30/07/2015 14:54:31 by RG

---

Area Percent Report
---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak RetTime Type Width Area Height Area # [min] [min] [mAU*s] [mAU] %
--- | ------ | ------ | ------ | ------ | ------ | ------ | ------ |
1 16.609 VB 0.8562 7.29081e4 1178.86538 84.5605
2 24.370 VB 1.0945 1.33227e4 174.59250 15.4395

Totals : 8.628999e4 1353.25888

---

S32
ADH (98:02) 1 ML/MIN

Injection Date : 29/01/2015 17:31:03
Sample Name : RG1509RAC  Location : Vial 21
Acq. Operator : RG
Acq. Instrument : HPLC-GPC  Inj Volume : 5 μl
Acq. Method : C:\ \METHODS\A0.M
Last changed : 29/01/2015 18:10:46 by RG
(modified after loading)
Analysis Method : C:\ \METHODS\RG.M
Last changed : 23/07/2015 18:17:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254, Ref=160,100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
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</thead>
<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*μ]</td>
<td>[μμμ]</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>40.899</td>
<td>0.055</td>
<td>1.33116e4</td>
<td>151.79311</td>
<td>49.9934</td>
</tr>
<tr>
<td>2</td>
<td>44.326</td>
<td>0.134</td>
<td>1.33203e4</td>
<td>143.03912</td>
<td>50.0066</td>
</tr>
</tbody>
</table>

Totals : 2.66367e4 294.83223
3b

ADH (98.02) 1 ML/MIN

Injection Date: 29/01/2015 18:18:36
Sample Name: RG1508
Acq. Operator: KG
Acq. Instrument: HPLC-GPC
Acq. Method: C:\HPCHEM\1\METHODS\AO.M
Last changed: 29/01/2015 18:10:46 by RG
(modified after loading)
Analysis Method: C:\HPCHEM\1\METHODS\RG.M
Last changed: 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTIs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>41.675</td>
<td>BB</td>
<td>1.125</td>
<td>2.49185e4</td>
<td>288.80475</td>
<td>88.8940</td>
</tr>
<tr>
<td>2</td>
<td>45.025</td>
<td>BB</td>
<td>1.1067</td>
<td>3113.22119</td>
<td>33.17999</td>
<td>11.1060</td>
</tr>
</tbody>
</table>
Injection Date : 02/02/2015 13:51:09   Seq. Line : 1
Sample Name : RG1511R2    Location : Vial 1
Acq. Operator : RG    Inj : 1
Acq. Instrument : HPLC-GPC    Inj Volume : 8 μl
Different Inj Volume from Sequence !  Actual Inj Volume : 5 μl
Acq. Method : C:\HPCHEM\1\METHODS\AO.M
Last changed : 02/02/2015 13:50:07 by RG
Analysis Method : C:\HPCHEM\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254.4 Ref=360.100

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21.437 FV</td>
<td>0.7369</td>
<td>6.24755e4</td>
<td>1263.40625</td>
</tr>
<tr>
<td>2</td>
<td>23.126 VB</td>
<td>0.6448</td>
<td>6.44013e4</td>
<td>1284.89001</td>
</tr>
</tbody>
</table>

Totals : 1.26878e5  2546.29626
ADH, 95:5 (Hx:iPrOH), 1 mL/min, GPC

Injection Date: 02/02/2015 16:15:32
Sample Name: RG1510BIS
Location: Vial 2
Acq. Operator: KG
Acq. Instrument: HPLC-GPC
Inj Volume: 8 µL
Acq. Method: C:\HPCHEM\1\METHODS\AO.M
Last changed: 02/02/2015 16:16:26 by RG
(modified after loading)
Analysis Method: C:\HPCHEM\1\METHODS\RG.M
Last changed: 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DADL A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area [min]</th>
<th>Height</th>
<th>Area [mAU*sec]</th>
<th>Area [mAU]</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>21.365</td>
<td>YV</td>
<td>0.7080</td>
<td>8.34426e4</td>
<td>1502.34717</td>
<td>79.9436</td>
<td>1502.34717</td>
<td>79.9436</td>
</tr>
<tr>
<td>22.724</td>
<td>VB</td>
<td>0.7256</td>
<td>2.09342e4</td>
<td>403.30258</td>
<td>20.0564</td>
<td>403.30258</td>
<td>20.0564</td>
</tr>
</tbody>
</table>

Totals: 1.04377e5 1905.64975
ODH1 95/05 1 ml/min HPLC

Injection Date: 15/09/2015 10:22:53
Sample Name: RG1513BI
Acq. Operator: RG
Acq. Instrument: HPLC 2
Inj Volume: 5 µl

Analysis Method: C:\HPCHEM\2\METHODS\C1.M
Last changed: 15/09/2015 10:16:31 by RG
(modified after loading)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 B, Sig=240,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>38.414</td>
<td>PB</td>
<td>1.4302</td>
<td>1.08117e4</td>
<td>109.09745</td>
<td>50.9591</td>
</tr>
<tr>
<td>2</td>
<td>44.026</td>
<td>PB</td>
<td>1.5452</td>
<td>1.04047e4</td>
<td>82.28828</td>
<td>49.0409</td>
</tr>
</tbody>
</table>

Totals: 2.12164e4 191.38573
ODH 95/05 1 ml/min HPLC

Injection Date: 15/09/2015 11:18:15
Sample Name: RG1781B
Acq. Operator: RG
Acq. Instrument: HPLC 2
Acq. Method: C:\HPCHEM\2\METHODS\C1.M
Last changed: 15/09/2015 10:10:31 by RG
(modified after loading)
Analysis Method: C:\HPCHEM\2\METHODS\C1.M
Last changed: 14/09/2015 16:24:05 by JW

DAD1 B, Sig=240,16 Ref=360,100 (RG/RG1781BLD)

Area Percent Report

Signal 1: DAD1 B, Sig=240,16 Ref=360,100

Peak RetTime Type Width Area Height Area %
# [min] | [min] | [AU*s] | [AU] | %
1 36.860 PB 1.5239 9.61367e4 854.92126 83.7728
2 43.044 BB 1.8415 1.86222e4 138.07594 16.2272
Totals: 1.14759e5 992.99721
AS, 92:08 (Hx:iPrCH), 1 mL/min, GPC

Injection Date : 06/02/2015 17:30:47
Sample Name : RG151592              Location : Vial 1
Acq. Operator : RG
Acq. Instrument : HPLC-GPC        Inj Volume : 8 μl
Acq. Method : C:\HEC\METHODS\RG.M
Last changed : 06/02/2015 17:48:29 by RG
(modified after loading)
Analysis Method : C:\HEC\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 8.464 VV</td>
<td></td>
<td>0.4901</td>
<td>1.28705e4</td>
<td>410.17987</td>
<td>47.3647</td>
</tr>
<tr>
<td>2 10.386 VB</td>
<td></td>
<td>0.5589</td>
<td>1.43026e4</td>
<td>335.82611</td>
<td>52.6353</td>
</tr>
</tbody>
</table>
AS, 92:08 (Hx:iPrOH), 1 mL/min, GPC

Injection Date : 06/02/2015 17:52:42
Sample Name : RG151492 Location : Vial 2
Acq. Operator : RG
Acq. Instrument : HPLC-GPC Inj Volume : 8 µl
Acq. Method : C:\BPCHEM\1\METHODS\RG.M
Last changed : 06/02/2015 17:08:29 by RG
(modified after loading)
Analysis Method : C:\BPCHEM\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime Type Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [nAU]</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 8.572 VB</td>
<td>0.3595</td>
<td>6739.86865</td>
<td>289.59125 87.6063</td>
</tr>
<tr>
<td>2 10.806 MM</td>
<td>0.3857</td>
<td>953.49225</td>
<td>41.20644 12.3937</td>
</tr>
</tbody>
</table>

Totals : 7693.36090 330.79769
AS, 98:02 (Hx:iPrOH), 1 mL/min, GPC

Injection Date : 09/01/2015 16:27:59
Sample Name : RG152798 Location : Vial 1
Acq. Operator : RG
Acq. Instrument : HPLC-GPC Inj Volume : 4 μL
Acq. Method : C:\HPChem\1\METHODS\RG.M
Last changed : 09/01/2015 16:25:01 by RG
(modified after loading)
Analysis Method : C:\HPChem\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20.426</td>
<td>BV</td>
<td>0.9955</td>
<td>7.57014e4</td>
<td>897.97900</td>
<td>47.7798</td>
</tr>
<tr>
<td>2</td>
<td>25.145</td>
<td>MM</td>
<td>1.7307</td>
<td>8.27365e4</td>
<td>796.77527</td>
<td>52.2202</td>
</tr>
</tbody>
</table>
AS, 98:02 (Hex:1PrOH), 1 mL/min, GPC

Injection Date : 09/02/2015 17:31:34
Sample Name : RG152598 Location : Vial 2
Acq. Operator : RG
Acq. Instrument : HPLC-GPC
Acq. Method : C:\HPCHEM\1\METHODS\RG.M
Last changed : 09/02/2015 16:25:03 by RG
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

| Peak RetTime Type Width Area Height Area |
|---|--|---|---|---|---|---|
| # | [min] | [min] | [mAU's] | [mAU] | % |
| 1 | 20.793 | 0.528 | 369.73752 | 8.39188 | 19.2121 |
| 2 | 24.436 | 0.6408 | 1654.76221 | 19.40345 | 80.7870 |

Totals : 1924.49973 27.79534
AS, 90:10, 1

Injection Date : 02/09/2015 14:04:14
Sample Name : RG1532F
Location : Vial 2
Acq. Operator : RG
Inj Volume : 4 µl
Acq. Instrument : HPLC 2
Acq. Method : C:\HPCHEM2\METHODS\C5.M
Last changed : 02/09/2015 14:03:00 by RG
(modified after loading)
Analysis Method : C:\HPCHEM2\METHODS\C5.M
Last changed : 21/04/2015 18:07:24 by JW

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/µl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area%</th>
</tr>
</thead>
<tbody>
<tr>
<td>11.803</td>
<td>VV</td>
<td>0.4351</td>
<td>3.77797e4</td>
<td>1269.69678</td>
<td>51.8064</td>
</tr>
<tr>
<td>13.810</td>
<td>VB</td>
<td>0.4934</td>
<td>3.51451e4</td>
<td>1047.60461</td>
<td>48.1936</td>
</tr>
</tbody>
</table>

Totals : 7.29248e4 2317.30139
AS 90/10 HEX/IPA 1ml/min HPLC2

Injection Date : 11/02/2015 16:41:58
Sample Name : RG31531 Location : Vial 41
Acq. Operator : RG
Acq. Instrument : HPLC 2
Acq. Method : C:\HPChem\2\METHODS\C6_1_50.M
Inj Volume : 8 μl
Last changed : 11/02/2015 16:29:08 by RG
(modified after loading)
Analysis Method : C:\HPChem\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

---

Area Percent Report

---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.000000 [ng/μl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

<table>
<thead>
<tr>
<th>Peak Ret Time</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
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<tbody>
<tr>
<td>#</td>
<td>[min]</td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
<td>%</td>
</tr>
<tr>
<td>1</td>
<td>11.539</td>
<td>VB</td>
<td>0.4608</td>
<td>3.81708e4</td>
<td>1234.12549</td>
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<tr>
<td>2</td>
<td>14.342</td>
<td>BV</td>
<td>0.4740</td>
<td>8415.80859</td>
<td>271.20303</td>
</tr>
</tbody>
</table>

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S44
IA 95/05 1.0 ml/min HPLC2

Injection Date : 08/09/2015 10:36:56
Sample Name : RG153SHY
Acq. Operator : RG
Acq. Instrument : HPLC 2
Acq. Method : C:\HPCHEM2\METHODS\Cl.m
Last changed : 08/09/2015 10:35:54 by RG
(modified after loading)
Analysis Method : C:\HPCHEM2\METHODS\Cl.m
Last changed : 03/06/2015 20:03:47 by RG

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

| Peak RetTime Type Width Area  Height Area % |
|----|----------|----------|----------|--------|
| 1  | 10.282 BB | 0.2108 | 267.14706 | 19.38325 | 50.6668 |
| 2  | 11.364 BB | 0.2484 | 259.90768 | 16.26430 | 49.3332 |
| Totals : |       |      | 527.05475 |       | 35.64755 |

S45
IA 95/05 1.0 ml/min HPLC2

Injection Date: 08/09/2015 12:25:55
Sample Name: RG1542HY
Location: Vial 3
Acq. Operator: RG
Acq. Instrument: HPLC 2
Acq. Method: C:\HPChem\2\METHODS\Cl.M
Last changed: 08/09/2015 11:55:25 by RG
(modified after loading)
Analysis Method: C:\HPChem\2\METHODS\Cl.M
Last changed: 03/06/2015 20:03:47 by RG

DAD 1 E, Sig=280,16 Ref=360,100 (RG/RG1542HY.D)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD 1 E, Sig=280,16 Ref=360,100

Peak Ret Time Type Width Area Height Area %
---|-------|---|-------|---|---|---|
1 10.529 PV 0.6806 2249.93433 51.40871 13.4973
2 12.563 VB 0.9250 1.4419564 234.62718 86.5027

Totals: 1.6669564 286.03589
AS, 90:10, 1

Injection Date : 02/09/2015 13:04:28
Sample Name : RO1534
Acq. Operator : RG
Acq. Instrument : HPLC 2
Acq. Method : C:\HPChem\2\METHODS\C5.M
Last changed : 02/09/2015 13:02:08 by RG
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\C5.M
Last changed : 21/04/2015 18:07:24 by JW

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.355</td>
<td>4.622</td>
<td>1529.2</td>
<td>49.311</td>
</tr>
<tr>
<td>2</td>
<td>10.942</td>
<td>4.752</td>
<td>1405.3</td>
<td>50.688</td>
</tr>
</tbody>
</table>

Totals : 9.37515e4 2934.54810
AS 90/10 1.0 ml/min HPLC

Injection Date : 04/09/2015 12:25:15
Sample Name : RG1533Q
Location : Vial 1
Acq. Operator : RG
Acq. Instrument : HPLC 2
Inj Volume : 8 µl

Acq. Method : C:\HPChem\2\METHODS\CS.M
Last Changed : 04/09/2015 12:28:11 by JW
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\CS.M
Last Changed : 02/09/2015 14:39:07 by RG

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type Width Area Height Area
# [min] [min] [nAU*sec] [nAU] %
----- | ------ | ------ | -------- | ---- |
1 9.523 VW 0.3671 4.0464884 1260.06921 79.5226
2 11.277 VB 0.3642 1.0413484 436.82479 20.4674

Totals : 5.0878364 1796.69400
HPLC 2 ADH 90/10 1.0ml/min

Injection Date : 23/06/2014 10:35:13
Sample Name : lsrac
Acq. Operator : ls
Acq. Instrument : HPLC 2
Acq. Method : C:\HPCHEM\2\METHODS\C5.M
Last changed : 23/06/2014 10:10:06 by ls
(modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C4.M
Last changed : 30/07/2015 14:54:31 by RG

DAD1 A, Sig=254.4 Ref=360.100 (LS/LSRAC.D)

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [mAUs]</th>
<th>Height [mAUs]</th>
<th>Area [%]</th>
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<tbody>
<tr>
<td>1 16.117</td>
<td>VB</td>
<td>0.3040</td>
<td>917.96014</td>
<td>46.17144</td>
<td>34.0357</td>
</tr>
<tr>
<td>2 19.305</td>
<td>BB</td>
<td>0.3866</td>
<td>948.96857</td>
<td>37.07191</td>
<td>35.1854</td>
</tr>
<tr>
<td>3 21.081</td>
<td>BB</td>
<td>0.3955</td>
<td>411.67584</td>
<td>15.82568</td>
<td>15.2639</td>
</tr>
<tr>
<td>4 27.916</td>
<td>BB</td>
<td>0.4854</td>
<td>418.44864</td>
<td>12.10217</td>
<td>15.5150</td>
</tr>
</tbody>
</table>

Totals : 2697.05319 111.17120
HPLC 2 ADH 90/10 1.0 ml/min

---

Injection Date: 20/06/2014 14:44:49
Sample Name: LS95
Acq. Operator: ls
Acq. Method: C:\HPCHEM\2\METHODS\C5.M
Last changed: 20/06/2014 12:38:25 by ls
Analysis Method: C:\HPCHEM\2\METHODS\C4.M
Last changed: 30/07/2015 14:54:31 by RG

---

Area Percent Report

---

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
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<td>15.010</td>
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<td>0.3108</td>
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<td>124.82218</td>
<td>13.8390</td>
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<tr>
<td>2</td>
<td>17.472</td>
<td>BB</td>
<td>0.3487</td>
<td>1993.60449</td>
<td>87.22513</td>
<td>10.7068</td>
</tr>
<tr>
<td>3</td>
<td>19.078</td>
<td>BB</td>
<td>0.3686</td>
<td>403.98499</td>
<td>16.91656</td>
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<tr>
<td>4</td>
<td>25.066</td>
<td>BB</td>
<td>0.5048</td>
<td>1.36455e4</td>
<td>420.21802</td>
<td>73.2845</td>
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</table>

Totals: 1.86199e4 649.18189
AD, 90:10 (Hx:1PrOH), 0.5 mL/min, HPLC2

Injection Date: 06/03/2015 10:31:11
Sample Name: RG1554 Location: Vial 41
Acq. Operator: RG
Acq. Instrument: HPLC 2 Inj Volume: 7 µl
Acq. Method: C:\HPChem\2\METHODS\C4.M
Last changed: 06/03/2015 9:52:40 by RG
(modified after loading)
Analysis Method: C:\HPChem\1\METHODS\RG.M
Last changed: 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Sample Amount: 1.00000 [ng/µl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210.8 Ref=360,100

<table>
<thead>
<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 13.977 VV</td>
<td></td>
<td>0.3773</td>
<td>1.28868e4</td>
<td>519.75317</td>
<td>8.5216</td>
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<tr>
<td>2 16.248 VV</td>
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<td>0.4459</td>
<td>1.34068e4</td>
<td>447.05405</td>
<td>8.8655</td>
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<tr>
<td>3 20.604 VB</td>
<td></td>
<td>0.5956</td>
<td>6.03721e4</td>
<td>1360.78552</td>
<td>39.9222</td>
</tr>
<tr>
<td>4 26.268 VB</td>
<td></td>
<td>0.7753</td>
<td>6.45588e4</td>
<td>1204.58386</td>
<td>42.6907</td>
</tr>
</tbody>
</table>

Totals: 1.51225e5 3532.17661
AD, 90:10 (Hx:iPrOH), 0.5 mL/min, HPLC2

Injection Date : 06/03/2015 12:25:55
Sample Name : RG1555 Location : Vial 42
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 7 µl
Acq. Method : C:\HPCHEM\2\METHODS\C4.M
Last changed : 06/03/2015 9:52:40 by RG
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\RG.M
Last changed : 02/07/2015 18:37:52 by DZ

Area Percent Report

Signal 1: DAD1 C, Sig=210,8 Ref=360,100 (C:\HPCHEM2\DATA\RG1555.D)

<table>
<thead>
<tr>
<th>Peak</th>
<th>Ret Time</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area%</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>13.917</td>
<td>VV</td>
<td>0.4083</td>
<td>7262.95410</td>
<td>259.68967</td>
<td>3.9818</td>
</tr>
<tr>
<td>2</td>
<td>16.160</td>
<td>VV</td>
<td>0.4792</td>
<td>3.28922e4</td>
<td>1033.71313</td>
<td>18.0327</td>
</tr>
<tr>
<td>3</td>
<td>20.532</td>
<td>VB</td>
<td>0.5588</td>
<td>1.06572e4</td>
<td>287.26599</td>
<td>5.8426</td>
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<tr>
<td>4</td>
<td>26.257</td>
<td>MM</td>
<td>1.2976</td>
<td>1.31591e5</td>
<td>1690.16077</td>
<td>72.1428</td>
</tr>
</tbody>
</table>

Totals : 1.82403e5 3270.82956
ODH, 95:05 (Hx:iPrOH), 1 mL/min, HPLC2

Injection Date : 06/03/2015 18:38:54
Sample Name : RG1556 Location : Vial 1
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 7 μl
Acq. Method : C:\HPCHEM\2\METHODS\C6.1_60.M Last changed : 06/03/2015 18:37:53 by RG
(modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C4.M Last changed : 30/07/2015 14:54:31 by RG

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1 : DAD1 E, Sig=280,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak #</th>
<th>RetTime</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>6.474</td>
<td>0.2046</td>
<td>5223</td>
<td>176.75989</td>
<td>36.1548</td>
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<tr>
<td>2</td>
<td>7.946</td>
<td>0.2651</td>
<td>1768</td>
<td>98.67942</td>
<td>25.3452</td>
</tr>
<tr>
<td>3</td>
<td>8.566</td>
<td>0.3247</td>
<td>1463</td>
<td>64.48297</td>
<td>20.9623</td>
</tr>
<tr>
<td>4</td>
<td>10.389</td>
<td>0.3715</td>
<td>1224</td>
<td>47.04834</td>
<td>17.5377</td>
</tr>
</tbody>
</table>

Totals : 6979.62329 386.97062
ODH, 95:05 (Hx:iPrOH), 1 mL/min, HPLC2

Injection Date : 06/03/2015 19:03:48
Sample Name : RG1557 Location : Vial 2
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 7 µl
Acq. Method : C:\HPCHM\2\METHODS\C6_1_60.M
Last changed : 06/03/2015 18:37:53 by RG
(modified after loading)
Analysis Method : C:\HPCHM\2\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

DAD1 D, Sig=230,16 Ref=360,100 (C:\HPCHM\2\DATA\RG\RG1557.D)

==================================================================
Area Percent Report
==================================================================
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.000000 [ng/µl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak #</th>
<th>RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
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<tbody>
<tr>
<td>1</td>
<td>6.470 VV</td>
<td>0.1810</td>
<td>2644.22144</td>
<td>212.68977</td>
<td>8.5163</td>
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<td>7.951 VV</td>
<td>0.3194</td>
<td>3620.89087</td>
<td>161.58305</td>
<td>11.6618</td>
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<tr>
<td>3</td>
<td>8.542 VB</td>
<td>0.4799</td>
<td>1.92941e4</td>
<td>621.99628</td>
<td>62.1406</td>
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<tr>
<td>4</td>
<td>10.620 BB</td>
<td>0.3961</td>
<td>5489.87207</td>
<td>203.91116</td>
<td>17.6813</td>
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</tbody>
</table>

Totals : 3.10491e4 1200.18027

anti-3I
55% ee
ADH, 90:10 (Rt:iPrOH), 0.3 mL/min, HPLC2

Injection Date : 06/03/2015 13:26:27
Sample Name : RG1558 Location : Vial 51
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 7 µl
Acq. Method : C:\HPCHM\2\METHODS\CS.M Last changed : 06/03/2015 13:30:27 by RG
(modified after loading)
Analysis Method : C:\HPCHM\1\METHODS\RG.M Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

<table>
<thead>
<tr>
<th>Sorted By</th>
<th>Signal</th>
<th>Multiplier</th>
<th>Dilution</th>
<th>Sample Amount</th>
<th>[ng/µl] (not used in calc.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use Multiplier &amp; Dilution Factor with ISTIDs</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Signal 1: DAD1 C, Sig=210.8 Ref=360,100

| Peak RetTime Type Width Area Height Area |
|---|---|---|---|---|---|---|
| #  | [min] | [min] | [mAU*s] | [mAU] | % |
| 1  | 28.730 | 0.4849 | 5.92133e+4 | 1492.39417 | 16.9219 |
| 2  | 32.883 | 0.5320 | 6.26863e+4 | 1411.87695 | 17.9144 |
| 3  | 42.394 | 1.1800 | 1.11811e+5 | 1579.30969 | 31.9533 |
| 4  | 47.060 | 1.2313 | 1.16210e+5 | 1573.00964 | 33.2103 |

Totals : 3.49921e5 6056.59045
ADH, 90:10 (H2O:iPrOH), 0.3 mL/min, HPLC2

Injection Date : 06/03/2015 17:03:11
Sample Name : RG1559  Location : Vial 62
Acq. Operator : RG  Inj Volume : 7 μL
Acq. Method : C:\HPChem\2\METHODS\C5.M
Last changed : 06/03/2015 17:00:01 by RG
(modified after loading)
Analysis Method : C:\HPChem\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.000000 [μg/μL] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>VB</td>
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<td>2</td>
<td>32.898</td>
<td>VV</td>
<td>0.5943</td>
<td>7.24003e4</td>
<td>1460.62329</td>
<td>23.7018</td>
</tr>
<tr>
<td>3</td>
<td>42.600</td>
<td>VB</td>
<td>0.7129</td>
<td>6.36760e4</td>
<td>1148.32288</td>
<td>20.8457</td>
</tr>
<tr>
<td>4</td>
<td>47.297</td>
<td>MM</td>
<td>1.5151</td>
<td>1.40113e5</td>
<td>1541.51941</td>
<td>45.8748</td>
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</tbody>
</table>

Totals : 3.05464e5 5006.21985
RAC-3n

ADH, 90:10, 1 mL/min, GPC

Injection Date: 15/10/2015 10:12:05  Seq. Line: 1
Sample Name: RGSV300RAC  Location: Vial 1
Acq. Operator: RG  Inj: 1
Acq. Instrument: HPLC-GPC  Inj Volume: 8 µl
Method: C:\HPCHEM\1\METHODS\STANDARD.M
Last changed: 15/10/2015 10:11:15 by RG

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

<table>
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<tr>
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<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 29.580 BB</td>
<td>0.6447</td>
<td>2450.37231</td>
<td>47.94058</td>
<td>16.8474</td>
<td></td>
</tr>
<tr>
<td>2 39.034 BB</td>
<td>0.9630</td>
<td>5125.66553</td>
<td>72.13313</td>
<td>35.0379</td>
<td></td>
</tr>
<tr>
<td>3 59.359 MM</td>
<td>1.6634</td>
<td>2298.46167</td>
<td>23.02680</td>
<td>15.7387</td>
<td></td>
</tr>
<tr>
<td>4 69.219 BB</td>
<td>1.3573</td>
<td>4719.40820</td>
<td>40.80551</td>
<td>32.3161</td>
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</tr>
</tbody>
</table>

Totals: 1.46039e4 183.90912
AD 90/10, 1 ML/MIN GPC

Injection Date : 13/10/2015 12:08:17
Sample Name : RG1793Q
Location : Vial 11
Acq. Operator : RG
Acq. Instrument : HPLC-GPC
Inj Volume : 8 ul
Acq. Method : C:\HPChem\1\METHODS\STANDARD.M
Last changed : 13/10/2015 12:07:19 by RG
(modified after loading)
Analysis Method : C:\HPChem\1\METHODS\STANDARD.M
Last changed : 15/10/2015 10:11:15 by RG

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
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<tbody>
<tr>
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<td>BV</td>
<td>0.6801</td>
<td>431.04910</td>
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<tr>
<td>2</td>
<td>38.639</td>
<td>BB</td>
<td>1.0224</td>
<td>981.55145</td>
<td>11.19002</td>
<td>28.8195</td>
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<tr>
<td>3</td>
<td>60.399</td>
<td>MM</td>
<td>2.1645</td>
<td>365.78699</td>
<td>2.81652</td>
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<tr>
<td>4</td>
<td>69.194</td>
<td>BB</td>
<td>1.6194</td>
<td>1627.46592</td>
<td>11.76825</td>
<td>47.7844</td>
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</table>

Totals : 3405.85446 33.34747
RAC-3n

AD, 96/04-1ML/MIN, GPC

Injection Date : 23/01/2015 11:02:07  Seq. Line : 1
Sample Name : RG1503  Location : Vial 1
Acq. Operator : RG  Inj. : 1
Acq. Instrument : HPLC-GPC  Inj Volume : 8 µl
Acq. Method : C:\HPChem\1\METHODS\AO.M
Last changed : 23/01/2015 10:59:47 by RG
Analysis Method : C:\HPChem\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by PZ

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 E, Sig=280,16 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
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</thead>
<tbody>
<tr>
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<td>VB</td>
<td>0.8802</td>
<td>1.95906e4</td>
<td>330.19925</td>
<td>25.4740</td>
</tr>
<tr>
<td>2</td>
<td>38.282</td>
<td>BB</td>
<td>1.3077</td>
<td>1.93565e4</td>
<td>196.08130</td>
<td>25.1695</td>
</tr>
<tr>
<td>3</td>
<td>48.782</td>
<td>BV</td>
<td>1.3990</td>
<td>1.87075e4</td>
<td>189.95593</td>
<td>24.3257</td>
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<td>52.005</td>
<td>VB</td>
<td>1.5218</td>
<td>1.92498e4</td>
<td>163.46898</td>
<td>25.0308</td>
</tr>
</tbody>
</table>

Totals : 7.69045e4  879.70546
AD, 96.04 (Hx:iPrOH), 1 mL/min, HPLC2

Injection Date : 05/03/2015 13:33:06
Sample Name : RG1561 Location : Vial 1
Acq. Operator : RG Acq. Method : C:\HPCHEM\2\METHODS\C4.M
Acq. Instrument : HPLC 2 Inj Volume : 7 µl
Last changed : 05/03/2015 12:37:15 by JW (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\RG.M
Last changed : 23/07/2015 18:37:52 by DZ

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Area Percent Report

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Sorted By : Signal Multiplier : 1.0000 Dilution : 1.0000 Sample Amount : 1.00000 [ng/ul] (not used in calc.) Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 E, Sig=280,16 Ref=360,100 (C:\HPCHEM\2\DATA\RG\RG1561.D)

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<tr>
<th>Peak RetTime</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>31.472 BB</td>
<td>1.0905</td>
<td>3.56025e4</td>
<td>494.20834</td>
<td>59.1082</td>
</tr>
<tr>
<td>2</td>
<td>46.024 BB</td>
<td>1.4078</td>
<td>4509.52588</td>
<td>43.84933</td>
<td>7.4868</td>
</tr>
<tr>
<td>3</td>
<td>55.905 BV</td>
<td>1.1228</td>
<td>1242.34949</td>
<td>13.21519</td>
<td>2.0626</td>
</tr>
<tr>
<td>4</td>
<td>58.916 VB</td>
<td>1.8951</td>
<td>1.88784e4</td>
<td>140.18692</td>
<td>31.3424</td>
</tr>
</tbody>
</table>

Totals : 6.02327e4 691.45979

---
RAC-3o

Injection Date : 16/03/2015 17:28:18
Sample Name : RG156699
Location : Vial 11
Acq. Operator : RG
Acq. Instrument : HPLC 2
Inj Volume : 10 μl
Acq. Method : C:\HPChem\2\METHODS\C5.M
Last changed : 16/03/2015 17:27:19 by RG
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\C4.M
Last changed : 30/07/2015 14:54:31 by RG
DAD1 A, Sig=254.4 Ref=360,100 (RG156699 D)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/μl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254.4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>Ret Time</th>
<th>Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area %</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>8.008</td>
<td>PP</td>
<td>0.1992</td>
<td>1013.5234</td>
<td>78.19730</td>
<td>13.8518</td>
</tr>
<tr>
<td>2</td>
<td>9.503</td>
<td>VB</td>
<td>0.2357</td>
<td>1029.51123</td>
<td>66.82724</td>
<td>14.0703</td>
</tr>
<tr>
<td>3</td>
<td>13.285</td>
<td>PB</td>
<td>0.4177</td>
<td>2072.16748</td>
<td>71.17280</td>
<td>28.3202</td>
</tr>
<tr>
<td>4</td>
<td>19.227</td>
<td>VV</td>
<td>0.7703</td>
<td>3201.72290</td>
<td>57.62107</td>
<td>43.7578</td>
</tr>
</tbody>
</table>

Totals : 7316.92395 273.81840
anti-3o

IA, 99:01 (Hx:iPrOH), 1 mL/min, HPLC2

---

Injection Date : 16/03/2015 17:54:56
Sample Name : RG156799
Acq. Operator : RG
Acq. Instrument : HPLC 2
Acq. Method : C:\HPChem\2\METHODS\C5.M
Last changed : 16/03/2015 17:27:19 by RG
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\C4.M
Last changed : 30/07/2015 14:54:31 by RG

---

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254.4 Ref=360,100

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] %
---|---------|--------|--------|--------|--------|
1 8.565 VV 0.2084 275.17932 20.53255 3.4004
2 10.405 VB 0.2716 2952.63647 169.17790 36.4863
3 14.080 BB 0.3181 4049.59961 193.55028 50.0417
4 20.555 BV 0.4899 815.03394 23.19522 10.0715

Totals : 8092.44934 406.45595
ADH 80/20 0.8 ml/min HPLC2

Injection Date : 22/09/2015 13:46:11
Sample Name : RO1?95RA Location : Vial 1
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 5 µl
(modified after loading)
Analysis Method : C:\HPChem\1\METHODS\CT100-C.M Last changed : 14/09/2015 11:40:40 by DA

--- Area Percent Report ---

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/µl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDS

Signal 1: DAD1 A, Sig=254,4 Ref=360,100

<table>
<thead>
<tr>
<th>Peak</th>
<th>Ret Time [min]</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area %</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>10.419</td>
<td>0.2253</td>
<td>6352.77002</td>
<td>427.59421</td>
<td>32.5624</td>
</tr>
<tr>
<td>2</td>
<td>11.683</td>
<td>0.2536</td>
<td>6440.04395</td>
<td>384.06607</td>
<td>28.0097</td>
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<tr>
<td>3</td>
<td>13.444</td>
<td>0.2848</td>
<td>5326.01099</td>
<td>177.34363</td>
<td>13.481</td>
</tr>
<tr>
<td>4</td>
<td>17.404</td>
<td>0.3821</td>
<td>3390.71191</td>
<td>135.43431</td>
<td>10.1499</td>
</tr>
</tbody>
</table>

Totals : 1.95095e4 1124.43822
ADH (80:20 Hex/iPrOH), 0.8 mL/min, HPLC2

Injection Date : 07/10/2015 18:10:27
Sample Name : RG1802QB
Acq. Operator : RG
Acq. Instrument : HPLC 2
Acq. Method : C:\HPChem\2\METHODS\C4.M
Last changed : 07/10/2015 17:18:19 by RG
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\C4.M
Last changed : 17/09/2015 16:22:39 by RG

Area Percent Report

Signal 1: DAD1 A, Sig=254,4 Ref=360,100
Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] %
---|-----|-----|-------|-------|---|
1 10.511 MM 0.2390 4416.16992 308.00412 12.5918
2 11.680 VB 0.2480 1.81144e4 1113.25611 51.6497
3 13.440 EV 0.2889 7710.00830 399.91135 21.9836
4 17.130 PP 0.3631 4831.07959 203.39536 13.7749
Totals : 3.50717e4 2023.57693
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with IS Fifo

Signal 1: DAD1 A, Sig=254.4 Ref=360.100

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width [min]</th>
<th>Area [mAU*sec]</th>
<th>Height [mAU]</th>
</tr>
</thead>
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<tr>
<td>#</td>
<td>[min]</td>
<td>[mAU*sec]</td>
<td>[mAU]</td>
</tr>
<tr>
<td>1</td>
<td>12.860</td>
<td>5.23954e4</td>
<td>1490.80823</td>
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<tr>
<td>2</td>
<td>15.213</td>
<td>1.60373e4</td>
<td>1358.99260</td>
</tr>
<tr>
<td>3</td>
<td>28.302</td>
<td>5377.10107</td>
<td>69.02440</td>
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<tr>
<td>4</td>
<td>35.715</td>
<td>5383.29834</td>
<td>56.99568</td>
</tr>
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Totals : 1.24659e5 2975.80106

Results obtained with enhanced integrator!

*** End of Report ***
ODH 90/10 1ML

Injection Date : 02/10/2015 12:33:30
Sample Name : RG1801Q Location : Vial 2
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 8 µl
Acq. Method : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed : 02/10/2015 11:54:49 by RG
(modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C6_1_60.M
Last changed : 15/09/2015 10:06:37 by JW

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig-254.4 Ref-360,100

| Peak RetTime Type Width Area Height Area | %     |
|-----|-------|------|--------|---------|-------|
| #   | [min] | [min] | [mAU*sec] | [mAU]  |       |
| 1   | 12.898| 0.5977| 7.02440e4 | 1798.39258 | 78.2072 |
| 2   | 15.702| 0.8335| 1.22000e4 | 215.81902  | 13.5917 |
| 3   | 28.722| 0.9333| 1.601.35779 | 20.49984  | 1.7840 |
| 4   | 36.024| 1.1219| 5715.10986 | 60.62152  | 6.3671 |

Totals : 8.97605e4 2095.33296
OH
OH
NO
2
RAC-5a

AD, 97:03 (Hx:iPrOH), 1 mL/min, HPLC2

============================================================================
Injection Date : 24/02/2015 13:47:02
Sample Name : RG154697 Location : Vial 1
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 7 µl
Acq. Method : C:\HPCHEN\2\METHODS\C4.M
Last changed : 24/02/2015 13:21:39 by RG
(modified after loading)
Analysis Method : C:\HPCHEN\1\METHODS\RG.M
Last changed : 33/07/2015 18:37:52 by DZ

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<table>
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<tr>
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<tr>
<td>40</td>
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<tr>
<td>60</td>
</tr>
<tr>
<td>80</td>
</tr>
<tr>
<td>100</td>
</tr>
</tbody>
</table>

-------------------------------------------------------------
Area Percent Report

============================================================================
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/µl] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig-210,9 Ref-360,100

| Peak RetTime Type Width | Area | Height | Area | %
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td># [min] [min] [mAU#s] [mAU]</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
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<tr>
<td>1</td>
<td>79.695</td>
<td>1.9132</td>
<td>4789.449092</td>
<td>29.41527</td>
</tr>
<tr>
<td>2</td>
<td>85.780</td>
<td>1.5671</td>
<td>3659.98535</td>
<td>27.45716</td>
</tr>
<tr>
<td>3</td>
<td>89.609</td>
<td>2.2634</td>
<td>1.83361e4</td>
<td>95.10253</td>
</tr>
<tr>
<td>4</td>
<td>94.529</td>
<td>2.6083</td>
<td>1.85267e4</td>
<td>83.25191</td>
</tr>
</tbody>
</table>

Totals : 4.53113e4 235.22688
ADH, 97:03 (Hx: iPrOH), 1 mL/min, HPLC2

Injection Date : 24/02/2015 17:56:25
Sample Name : E01527AMH
Location : Vial 2
Acq. Operator : RG
Acq. Instrument : HPLC 2
Inj Volume : 4 µl
Acq. Method : C:\HPChem\METHODS\C5.M
Last changed : 24/02/2015 17:55:18 by RG
(modified after loading)
Analysis Method : C:\HPChem\METHODS\RG.M
Last changed : 23/07/2015 10:37:52 by RG

Area Percent Report

Signal 1: DAD1 C, Sig-210, Ref-360, 100
Peak RetTime Type Width Area Height Area
---|-----|-----|-----|-----|-----|-----|-----|
1  79.323 BE  1.2701 2.36037e4  232.55391  19.9125
2  85.378 RV  1.1225  7002.76855  76.31704  6.6565
3  69.590 VE  1.4855  8.25615e4  636.31848  69.6459
4  94.456 BE  1.4584  4476.69727  36.10831  3.7764
Totals : 1.16545e5 1025.31775
AD, 97:03, 1 mL/min, GPC

Injection Date: 09/03/2015 13:32:40  Seq. Line: 1
Sample Name: EG154997  Location: Vial 11
Acq. Operator: RG  Inj: 1
Acq. Instrument: HPLC-GPC  Inj Volume: 7 µl
Different Inj Volume from Sequence! Actual Inj Volume: 9 µl
Acq. Method: C:\HPChem\METHODS\RG.M
Last changed: 09/03/2015 13:31:32 by RG
Analysis Method: C:\HPChem\METHODS\RG.M
Last changed: 23/07/2015 18:37:52 by EZ

---

Area Percent Report
---

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig-230,16 Ref-360,100

<table>
<thead>
<tr>
<th>Peak Ret Time Type</th>
<th>Width [min]</th>
<th>Area [nAU*s]</th>
<th>Height [nAU]</th>
<th>Area [%]</th>
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</thead>
<tbody>
<tr>
<td>1 23.674 VB</td>
<td>0.7621</td>
<td>7822.99882</td>
<td>148.13287</td>
<td>9.8614</td>
</tr>
<tr>
<td>2 32.580 BE</td>
<td>1.2633</td>
<td>1.63134e4</td>
<td>186.36140</td>
<td>20.5641</td>
</tr>
<tr>
<td>3 39.397 VB</td>
<td>1.2937</td>
<td>2.97849e4</td>
<td>326.07205</td>
<td>37.5459</td>
</tr>
<tr>
<td>4 61.133 BR</td>
<td>1.5638</td>
<td>2.54081e4</td>
<td>104.78745</td>
<td>12.0286</td>
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</tbody>
</table>

Totals: 7.93293e4 855.35378
AD, 97:03, 1 mL/min, GPC

Injection Date: 09/03/2015 16:31:40
Sample Name: RG1548
Location: Vial 12
Acq. Operator: RG
Acq. Instrument: HPLC-GPC
Inj Volume: 7 µl
Acq. Method: C:\HPCEM\1\METHODS\RG.M
Last changed: 09/03/2015 13:31:32 by RG
Analysis Method: C:\HPCEM\2\METHODS\C4.M
Last changed: 03/07/2015 14:54:31 by RG

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

<table>
<thead>
<tr>
<th>#</th>
<th>RetTime [min]</th>
<th>Type</th>
<th>Width [min]</th>
<th>Area [mAU*sec]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
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</thead>
<tbody>
<tr>
<td>1</td>
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<td>VB</td>
<td>0.7498</td>
<td>1.538104</td>
<td>301.15872</td>
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</tr>
<tr>
<td>2</td>
<td>32.733</td>
<td>BB</td>
<td>1.3732</td>
<td>1.505544</td>
<td>140.16357</td>
<td>18.0389</td>
</tr>
<tr>
<td>3</td>
<td>39.147</td>
<td>BB</td>
<td>1.2436</td>
<td>4.782744</td>
<td>537.74420</td>
<td>57.3054</td>
</tr>
<tr>
<td>4</td>
<td>60.476</td>
<td>BB</td>
<td>1.4577</td>
<td>5196.83545</td>
<td>42.05306</td>
<td>6.2267</td>
</tr>
</tbody>
</table>

Total: 82490.88 1001 11056

S70
AD 97/03 1 ml/min HPLC2

Injection Date : 21/09/2015 12:51:28
Sample Name : RG1551V8 Location : Vial 51
Acq. Operator : RG
Acq. Instrument : HPLC 2

Acq. Method : C:\HPCHEM\2\METHODS\C3_1_60.M
Last changed : 21/09/2015 12:51:58 by RG
(modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\C3_1_60.M
Last changed : 30/03/2015 14:08:24 by RG

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig=230,16 Ref=360,100

<table>
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<tr>
<th>Peak</th>
<th>RetTime Type</th>
<th>Width [min]</th>
<th>Area [mAU*s]</th>
<th>Height [mAU]</th>
<th>Area [%]</th>
</tr>
</thead>
<tbody>
<tr>
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<td>20.021 VB</td>
<td>0.6149</td>
<td>1154.50528</td>
<td>28.12388</td>
<td>10.8056</td>
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<tr>
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<td>33.020 BV</td>
<td>0.9216</td>
<td>3920.74097</td>
<td>61.02012</td>
<td>36.6962</td>
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<tr>
<td>3</td>
<td>34.643 VB</td>
<td>1.0266</td>
<td>4547.98730</td>
<td>59.98472</td>
<td>42.5669</td>
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</table>

Totals : 1.06843e4 174.07660
AD 97/03 1 ml/min HPLC2

Injection Date : 21/09/2015 13:58:09
Sample Name : RG1550Puro-V7-20 Location : Vial 52
Acq. Operator : RG
Acq. Instrument : HPLC 2 Inj Volume : 7 µl
Acq. Method : C:\HPChem\2\METHODS\C3_1_60.M
Last changed : 21/09/2015 12:51:58 by RG
(modified after loading)
Analysis Method : C:\HPChem\2\METHODS\C3_1_60.M
Last changed : 30/03/2015 14:09:24 by RG

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [ng/ul] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 D, Sig-230,16 Ref-360,100

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area Width</th>
<th>Area Height</th>
<th>Area %</th>
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<tbody>
<tr>
<td></td>
<td>[min]</td>
<td>[mAU*s]</td>
<td>[mAU]</td>
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</tr>
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<tr>
<td>2</td>
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<td>0.6903</td>
<td>2654.88940</td>
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<tr>
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<td>32.847 BB</td>
<td>1.1166</td>
<td>2.4974364</td>
<td>335.43155</td>
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Totals : 3.24048e4 503.16233
References